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PROPERTIES OF A POLYURETHANE ELASTOMER

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ABSTRACT

The energy which is required to form a unit of new surface in the fracture of a polyurethane elastomer is determined. The rate sensitivity of the material has been reduced by swelling it in Toluene. This paper describes primarily the experimental work of measuring the lower limit of the fracture energy. Starting with this value and the creep compliance the rate dependence of the fracture energy for the unswollen material has been determined. It is thus shown that the dependence of the fracture energy on the rate of crack propagation is caused by the energy dissipation around the tip of the crack. Good agreement between the theoretically and experimentally determined relationship for the rate sensitive fracture energy is demonstrated.

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INTRODUCTION

In 1953 Rivlin and Thomas [1] demonstrated that a criterion for fracture of viscoelastic materials can be established which has the same form as the fracture criterion for elastic materials derived by Griffith [2] on the basis of energy conservation. Important, although not necessary [3], in this derivation is the concept of fracture energy*. Later it was shown by Greensmith and Thomas [4] that if this criterion is extended to propagating cracks the fracture energy becomes a monotonically increasing function of the rate of propagation. Figure 1 has been obtained from the data presented in Ref. 4 for a certain natural rubber vulcanizate and illustrates the rate dependence of the fracture energy.

Since the energy dissipation caused by the viscoelasticity of the material is included in the fracture energy the question arises immediately how much of the rate dependence of the fracture energy is solely attributable to viscous energy dissipation around the crack tip and how much, if any, rate sensitivity remains after this contribution has been subtracted.

In an effort to establish a lower bound of the fracture energy one is naturally led to investigate the fracture process at temperatures well above the glass transition temperature and crack velocities which approach zero. Experimentally this can be difficult with the problem primarily centered on the question whether the true limit below which

* Sometimes referred to as surface energy. We shall clarify the terminology later.

propagation is not possible has been reached or merely the limit of the experimentalist's patience. Under normal circumstances average rates of propagation can be reliably measured down to about 10^{-3} in/min, cf. Figure 1. The maximum test temperature is limited by the possibility of thermal degradation of the material. Within the frame of these limitations it may not be possible to observe experimentally a lower bound of the fracture energy. The data of Greensmith and Thomas, Figure 1, for instance, shows a slight tendency to level off at about $T_c = 10^{-0.3}$ lbs/in but it seems speculative to deduce from this data alone that there is a lower bound on T_c . The experimental data for another typical viscoelastic material shown in Figure 10 exhibits the same general behavior and does not allow a firmer conclusion with regard to a lower limit.

Inasmuch as the experimental determination of the lower limit of the fracture energy amounts to testing the material under conditions in which the energy dissipation caused by viscosity is negligibly small, it seems natural to try to remove the internal viscosity sufficiently by swelling the material in a suitable solvent and then performing fracture tests in this state. If the material becomes practically rate insensitive in the swollen state the fracture criterion for an elastic material may be applied and the fracture energy be calculated from experimental data. It is then only necessary to relate this value to its counterpart in fracture of the unswollen material.

It should be pointed out that the search for the lower limit of the fracture energy is of more than academic interest. The existence of a lower limit would guarantee that under certain long term loading conditions crack propagation would not be possible. With regard to

engineering applications a lower limit of the fracture energy would thus assure continual service of the structure without eventual failure due to crack propagation.

TERMINOLOGY

Before proceeding to the experimental determination of the fracture energy, it is appropriate to comment on the distinction between fracture and surface energy made in this paper. Depending on one's point of view the terminology of fracture or surface energy may or may not be interchangeable. On the one hand, the surface energy may be defined as the amount of work required to form a unit of new surface, regardless of the molecular processes taking place during the creation of the new surface. On the other hand some characteristics of the surface forming process may be introduced into the definition of the surface energy. In this sense one may distinguish between work done against intramolecular and intermolecular forces. The latter gives rise to the classical concept of surface tension in liquids. The disruption of chemical bonds would require additional work which may considerably increase the total work necessary to form a unit of new surface area. In the absence of energy dissipation by other mechanisms we shall refer to this total work as the intrinsic fracture energy of the material.

In view of this definition the energy which has to be supplied in order to form new surface at a given rate may then be called the rate dependent fracture energy T_c , or for short, just fracture energy. Inasmuch as a time dependent stress and strain field around the crack tip causes a certain crack velocity history or vice versa the fracture energy may also somehow depend on this history. We shall demonstrate that for constant crack velocity the fracture energy T_c ,

as a function of the magnitude of this constant velocity, can be calculated from a knowledge of the intrinsic fracture energy S and the creep compliance D_{cr} of the material, assuming the laws of linear viscoelasticity can be applied to adequately describe the material behavior. The rate dependent fracture energy can hence be considered as a deduced property instead of a fundamental property of the material.

EXPERIMENTAL DETERMINATION OF THE INTRINSIC FRACTURE ENERGY S

We shall first describe how the intrinsic fracture energy S can be determined by swelling the material in a suitable agent and performing fracture tests in this state. The result obtained in this manner will then be compared to the value calculated from experimental data obtained for the unswollen material at very low crack velocities and temperatures well above the glass transition temperature. The material chosen for this investigation is a polyurethane elastomer with the trade name Solithane 113. The particular composition employed in this program was prepared from equal amounts by volume of the two basic components resin and catalyst and will be referred to as Solithane 50/50. The mechanical properties of this material are documented in reference [5].

Since S can only be directly determined if the energy dissipation caused by viscosity is negligibly small compared to the magnitude of S we must first show to what degree swelling removes the internal viscosity of the material. The desired effect will be most pronounced if the volume increase by swelling is greatest without severing the integrity of the network. Figure 2 shows the volume increase of Solithane 50/50 in various poorly hydrogen bonded solvents listed in Table 1. The solubility parameter of Solithane 50/50 is seen to be between 9.5 and $10 \sqrt{\text{cal./cm}^3}$. Toluene, with $\delta = 8.9 \sqrt{\text{cal./cm}^3}$, was chosen on the basis of this result as a suitable swelling agent for our purpose. It does not yield the maximum volume increase but was considered the easiest solvent to work with out of the group which has solubility parameters near the one of Solithane 50/50.

The reduction of internal viscosity by swelling can be gauged by investigating the rate dependence of the stress-strain curve, of failure data, and of other material responses of the swollen material which exhibit the characteristics of viscoelastic behavior in the unswollen state. The device pictured in Figure 3 was designed for the tests in the swollen state. It consists in principal of a tank which is bolted on the Instron cross head. A spring loaded clamp is mounted on the tank bottom. Two parallel, vertical rods are part of the tank structure. These rods guide a carriage which holds the upper clamp and is connected to the Instron load cell. The carriage rides on ball bearings which can be accurately adjusted so as to achieve parallelism between lower and upper clamp which is very important for fracture tests on strip specimen. The displacement of the clamps was monitored by two linearly variable differential transformers in addition to the record obtained from the built-in Instron equipment. The clamps can accommodate specimen with length up to 6 in. and are easily operated by means of clamp opening cams. After the specimen had reached its swelling equilibrium it was inserted in this device and remained submerged for the whole length of the experiment. The test temperature could be adjusted to any level in the range from -5°C to 50°C by installing copper coils through which a cooled or heated mixture of ethyleneglycol and water was pumped.

Figures 4 and 5 show the uniaxial stress-strain response in form of Mooney-Rivlin plots for unswollen and swollen Solithane 50/50, which has a glass transition temperature T_g of about -20°C .

σ denotes the engineering stress and λ stands for the stretch ratio l/l_0 . The response of the unswollen material at -5°C is seen to be strongly dependent on the strain rate. The stress-strain curves for the swollen material at -2°C on the other hand are practically independent of the rate although it has been changed by a factor of 1000. This indicates a substantially reduced internal viscosity in the swollen material as compared to the unswollen material. The response in the swollen state is furthermore seen to be essentially neo-Hookean. Similar tests at higher temperatures also showed that the direct relationship between absolute temperature and the constant C_1 in the neo-Hookean constitutive law which is predicted by the classical theory of rubber elasticity [7] is observed in the swollen state within the limits of experimental error [5].

The effect of swelling becomes even more obvious when the failure properties as exemplified by the strain at rupture under uniaxial tension are compared. Figure 6 gives the strain at failure for unswollen Solithane 50/50 and exhibits a strong dependence on the strain rate or, via the time-temperature shift principle, on the temperature. The strain at failure is seen to vary from about 25% to roughly 200% depending on strain rate and temperature, whereas the failure strain of the swollen material exhibits hardly more than a statistical scatter about a certain mean value for all tested strain rates. The mean value seems to depend slightly on the test temperature but the data cannot be shifted as in the case of the unswollen material, see Figure 7.

Taking the examples just presented as sufficient evidence that the internal viscosity of the material is reduced to an acceptable level in the temperature range of interest we may proceed to perform fracture tests in the swollen state. These tests were carried out on specimen as shown in the insert of Figure 8. The specimen were cut out of sheets of cast Solithane 50/50 with a thickness of 1/32 in. in the unswollen state. An initial crack of the same length as the strip width was cut in the specimen center on one of the narrow edges. The long edges were clamped in the device already described and displaced at a constant rate. The crack becomes unstable at a certain strain and rapidly propagates through the sheet with a velocity which is several orders of magnitude higher than in the unswollen state [5].

Assuming the strip to behave perfectly elastic and the stresses around the crack tip to be equal to the ones in an infinitely long strip the following instability criterion can be derived from the stress-strain analysis of this geometry [8, 9] and for plane stress conditions:

$$\epsilon_{crit} = \sqrt{\frac{2S_{sw}(1-\nu^2)}{E_{sw}b}} \quad (1)$$

where ϵ_{crit} = critical strain at which the crack begins to propagate

S_{sw} = intrinsic fracture energy of the swollen material

E_{sw} = Young's Modulus of the swollen material

b = half strip width

ν = Poisson's ratio of the swollen material.

Only small strains $\epsilon_{crit}(\sim 2\%)$ are necessary to cause instability and the neo-Hookean material behavior is very well approximated by Hook's law at strains of this magnitude. As always in these problems there is some doubt about the material behavior at the crack tip but we assume the laws of elasticity to hold in this region, too. The stress analysis of a cracked, infinitely long strip shows that a sufficiently slender strip with a crack of about the same length as its width may be considered as a semi-infinite crack in an infinitely long strip [8,9]. Tests were run with strips of different aspect ratios to make sure the finite length of the strip had no effect on the end result.

Equation (1) furnishes a relationship through which the intrinsic fracture energy of the swollen material can be calculated from the strain at which instability occurs. Young's modulus was determined for each sheet by measuring the slope of the stress-strain curve at zero. Poisson's ratio is taken to be equal to 0.5. This assumption is not as unreasonable as it seems to be at first sight for a swollen material. According to the theory of rubber elasticity [7] the material is able to actually absorb more liquid when it is stretched. The time which is necessary to complete the diffusion process through which the additional liquid enters, however, is much longer than the length of any of the fracture tests that were performed [5,10] and the volume increase during the tests could be neglected.

The results of these tests are shown in Figures 8 and 9. The fracture energy of the swollen material is plotted there as a function of strain rate, aspect ratio of the strip, and temperature. It should be remembered that in the swollen state a distinction

between the intrinsic fracture energy and rate dependent fracture energy is not necessary because the material behaves in a brittle manner. Neither the strain rate nor the temperature seem to systematically affect the magnitude of S_{sw} in the range tested. The data points are scattered over a band ranging from about 0.06 lbs/in to 0.1 lbs/in. This band is seen to be very narrow compared to the large range of S which is obtained for the unswollen material at various crack velocities, cf. Figures 1,10. A certain amount of data scatter has to be expected because the condition of the initial crack tip is very important for the onset of fracture. The initial crack was cut with a razor blade and the shape of the crack tip could not be further controlled. Formula (1) from which S_{sw} has been calculated is based on the stress field for a line crack which has a sharper initial tip than the actual crack tip. The actual stresses at the crack tip will hence be slightly smaller than in theory and the values of S_{sw} calculated from (1) will be slightly higher depending on how much the actual shape of the tip differs from the one of a line crack. The lower limit of $S_{sw} = 0.06$ lbs/in will thus be the value that should come closest to the true value.

Having determined the fracture energy in the swollen state we must now estimate the effect of swelling on this quantity in order to derive the value of this energy in the unswollen material. We shall do this by assuming with Lake and Thomas [11] that the intrinsic fracture energy is essentially a measure of the chain bond strength only.

Consider a sheet of unit thickness to be submerged in a solvent. After swelling equilibrium has been reached the sheet thickness is R . We now extend a crack which was contained in the sheet by a small distance e . The work necessary to form the new surface is

$$e R S_{sw} = n_s E_b e R + 2 S_s \frac{e}{R} \quad (2)$$

where

n_s = number of polymer chains crossing a plane of size unity in the swollen material,

E_b = energy required to break a polymer chain, assumed to be unaffected by the presence of the swelling agent,

S_s = surface tension of the interface between swelling agent and polymer.

The last term in eq. (2) accounts for the increase in surface area of the liquid when the chains are broken. The material is assumed to be completely without voids in the unswollen state.

The number of polymer chains n passing through a unit area of unswollen material and the corresponding quantity for the unswollen material n_s are simply related by

$$n = n_s R^2. \quad (3)$$

According to our assumptions the intrinsic fracture energy of the unswollen material is equal to

$$S = n E_b = n_s E_b R^2 \quad (4)$$

and the following relationship can be obtained from (2)

$$S = R^2 S_{sw} - 2 S_s \quad (5)$$

The surface tension of the swelling agent may be assumed small (e.g., $S = 1.625 \times 10^{-4}$ lbs/in for Toluene in air at 20°C) compared to the quantity $R^2 S_{sw}$ and we have approximately

$$S = R^2 S_w. \quad (6)$$

For the Solithane 50/50-Toluene system a linear swelling ratio $R=1.39$ has been found and with the lower limit of S_{sw} one calculates a value of $S=0.116$ lbs/in for the unswollen material.

The results of fracture tests with unswollen Solithane 50/50 are presented in Figure 10. A long narrow strip was used again and the velocity of a crack propagating along the centerline measured as a function of strain [10]. From this data the rate dependent fracture energy T_c can be calculated by considering the strain energy release as the crack proceeds with constant velocity [4]. In case of a strip, T_c is given by

$$T_c = \frac{E_r \epsilon_o^2 b}{1 - \nu^2} \frac{T}{273} \quad (7)$$

where

- E_r = rubbery or long time modulus at $T = 273^\circ\text{K}$,
- ϵ_o = lateral strain,
- T = absolute temperature.

The data points shown in Figure 10 are obtained through application of (7) assuming the material to be incompressible. They are plotted on a reduced time scale. A comparison of the time-temperature shift

factors listed in this figure with the ones of Figure 6 shows a fairly good agreement in the temperature dependence.

Taking the value of T_c for the two lowest reduced velocities in Figure 10 as a measure of the intrinsic surface energy of Solithane 50/50 one obtains

$$S = \lim_{v \rightarrow 0} \frac{1}{2} T_c(v) = 0.08 \text{ lbs/in}$$

where v denotes the crack velocity. This value is slightly smaller than the one obtained from tests on the swollen material. Taking roughly the average of the values obtained by the two methods one arrives at the value

$$S = 0.1 \text{ lbs/in}^2 \pm 20\%$$

for the intrinsic fracture energy of Solithane 50/50.

Lake and Thomas [11] calculated theoretically a value

$$S = 2.0 \cdot 10^4 \text{ erg/cm}^2 = 0.114 \text{ lbs/in}$$

for vulcanized rubber. The network characteristics from which this value is calculated are to the authors knowledge unavailable for Solithane 50/50. But the order of magnitude of S may be assumed to be the same for both materials. The agreement between the above values is possibly coincidental but seemed worth mentioning.

For the system considered here the advantage of determining the intrinsic fracture energy S from tests in the swollen state is

easily recognized when the large range of T_c is considered in Figure 10. Depending on temperature and crack velocity the fracture energy may assume any value between roughly 0.16 and 16 lbs/in. A whole series of tests has to be run to determine the fracture energy-crack velocity relationship in order to be sure that S is calculated from data at the lower end of this curve. In the case of Solithane 50/50 a test in the swollen state, however, would yield a value for S which would at most differ by a factor of 3 from the correct value whereas only one test in the unswollen state may lead to a result which is off by an order of magnitude.

THE SIGNIFICANCE OF S IN VISCOELASTIC FRACTURE

We shall now consider the role which the intrinsic fracture energy S plays in the fracture of viscoelastic materials and in particular how it is related to the rate dependent fracture energy T_c , cf. Figure 1, 10. The theoretical development of this connection has been presented elsewhere [12] and we shall only quote the result necessary for the subsequent analysis and for the interpretation of experimental data. For the strip geometry already discussed, reference [12] asserts that for a linearly viscoelastic material the following relationship between lateral strain ϵ_o , crack velocity v , and absolute temperature T holds

$$\frac{2S}{E_r} \frac{1}{G(\frac{\Delta a}{v \varphi_t})} = \frac{\epsilon_o^2 E_r b}{(1-v^2)} \frac{T}{273} \quad (8)$$

where

$$G(t) = 2 \left[\frac{1}{t} \int_0^t D_{cr}(\tau) d\tau - \frac{1}{t^2} \int_0^t \tau D_{cr}(\tau) d\tau \right]$$

$D_{cr}(t)$ = creep compliance,

Δa = a length which is experimentally determined by fitting data, about 150 \AA for Solithane 50/50,

φ_T = time-temperature shift factor.

Comparing equations (7) and (8) leads immediately to the expression

$$T_c = \frac{2S}{E_r} \frac{1}{G(\frac{\Delta a}{v \varphi_T})} \quad (9)$$

which gives the fracture energy as a function of crack velocity and temperature in terms of a rate independent intrinsic fracture energy and a function G of the creep compliance of the material.

It is a simple matter to show that $G(\infty) = D_{cr}(\infty)$, and it follows that

$$T_c(o) = 2S. \quad (10)$$

The intrinsic fracture energy S is thus equal to half the energy which is required for rupture if there occurs no energy dissipation through viscous forces around the tip of the advancing crack. It appears reasonable, therefore, to assume that if the crack velocity varies the energy which needs to be supplied for the fracture process depends on the rate of change of the crack velocity, because the amount of dissipation around the crack tip depends on this rate of change.

We emphasize that the rate dependence of the fracture energy T_c arises solely from energy dissipation due to viscous forces which can be expressed by other material properties like the creep compliance D_{cr} . The quantity T_c is hence not an intrinsically rate dependent material property which only arises out of the fracture process. In order to underscore this statement we present in Figure 10 also a comparison between the calculated fracture energy T_c , according to (9), as a function of crack velocity v and the experimentally determined relationship between T_c and v for Solithane 50/50. The value of S which has been determined earlier and the function $G(t)$ shown in Figure 11 have been used in the calculation. The reciprocal relaxation modulus E_{rel}^{-1} and the creep compliance of Solithane 50/50 are also shown in this figure. It can be seen that the main difference between these functions is a shift along the time axis. The fracture

energy as a function of crack velocity is easily calculated from the experimental relationship between strain ϵ_0 and crack velocity v by considering the change in reversibly stored energy in the strip [1]. Good agreement between theoretical and experimental dependence of the fracture energy on the crack velocity in Solithane 50/50 is seen to exist over the whole range of velocities.

CONCLUSION

The fracture energy T_c which has to be supplied to propagate a crack with a certain velocity through a viscoelastic material is not an intrinsically rate dependent quantity. It can be calculated from the intrinsic fracture energy S which is a rate independent material property and the creep compliance D_{cr} of the material. Good agreement between theory and experiment has been demonstrated for a polyurethane elastomer. The intrinsic fracture energy can be quickly determined from fracture tests of the swollen material.

TABLE 1

Swelling Agents

Name	Solubility Parameter
	Ref. [6] $\sqrt{\text{cal./cm}^3}$
n-pentane	7.0
n-hexane	7.3
n-heptane	7.4
Methylcyclohexane	7.8
Cyclohexane	8.2
Carbontetrachloride	8.6
Toluene	8.9
Benzene	9.2
Chlorobenzene	9.5
o-dichlorobenzene	10.0
1-bromonaphtaline	10.6
Methylcellosolve	10.8
Acetonitrile	11.8
Nitromethane	12.7

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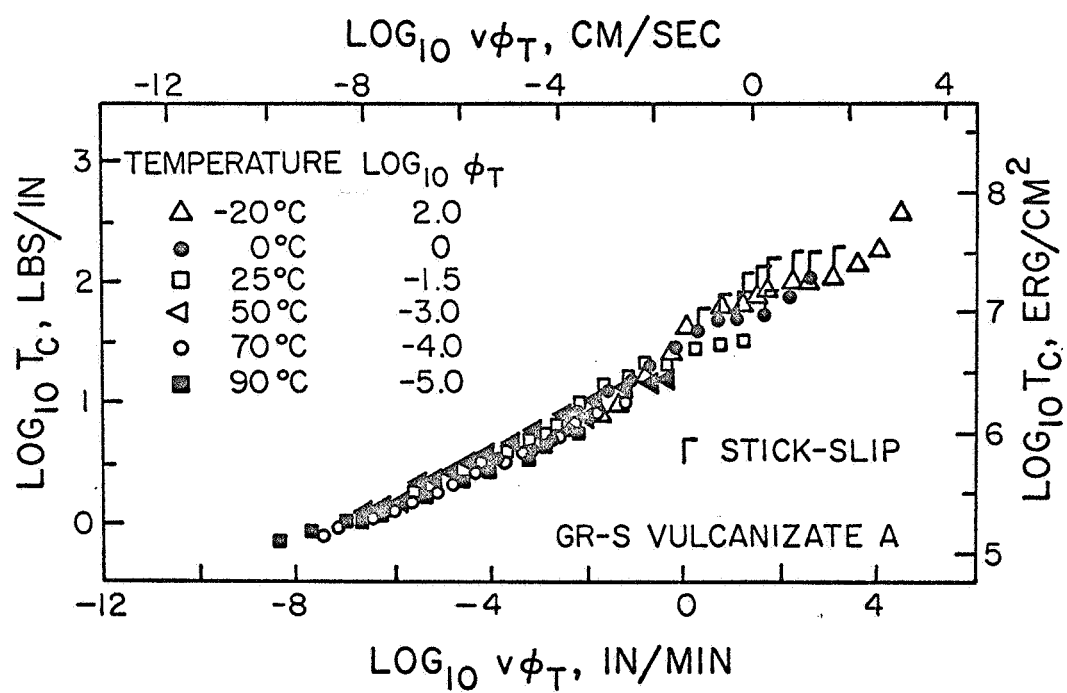


FIG.1 SHIFTED TEAR ENERGY DATA OF GREENSMITH AND THOMAS, Ref. 4

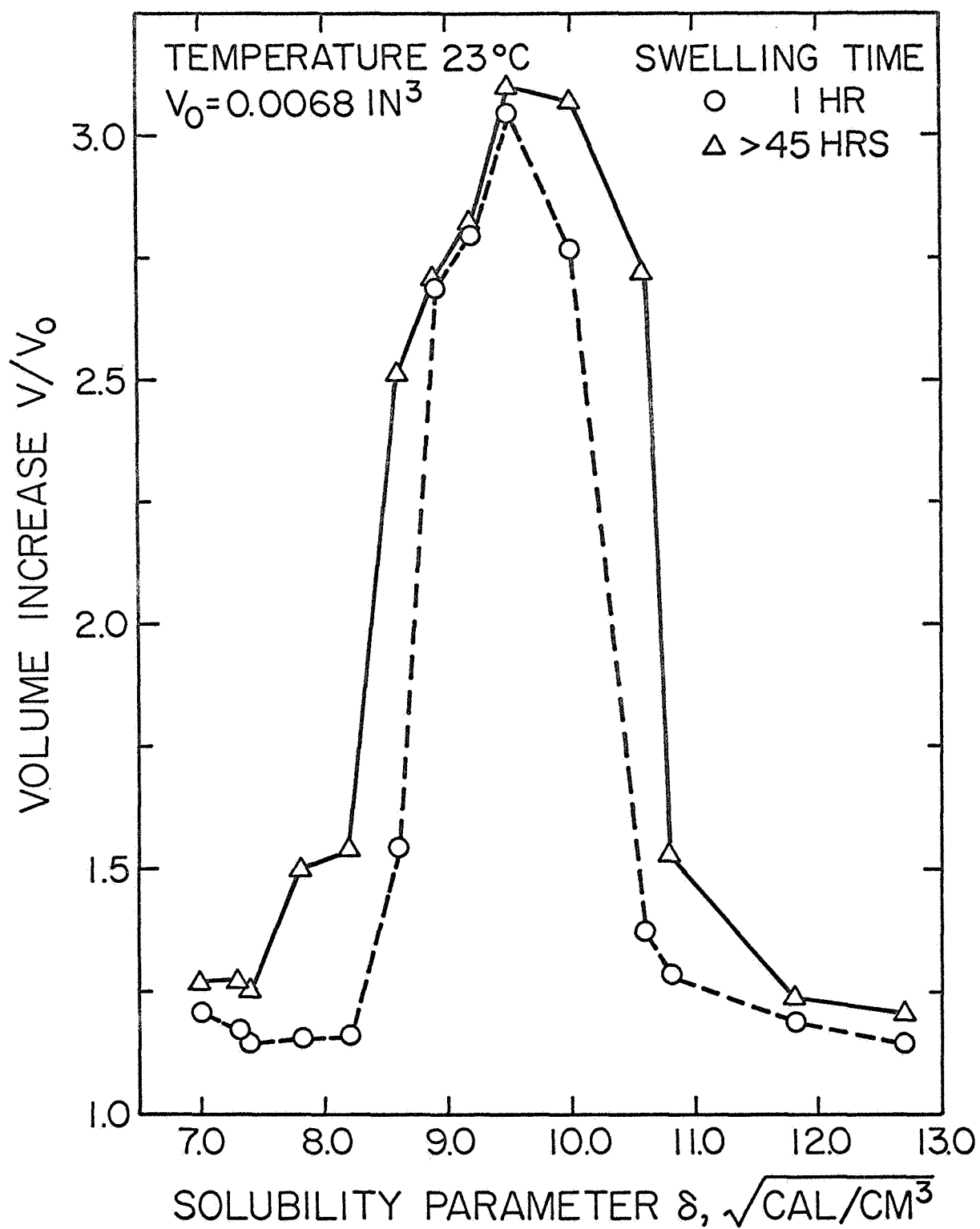


FIG.2 SWELLING RATIO OF SOLITHANE 50/50 IN POORLY HYDROGEN BONDED SOLVENTS

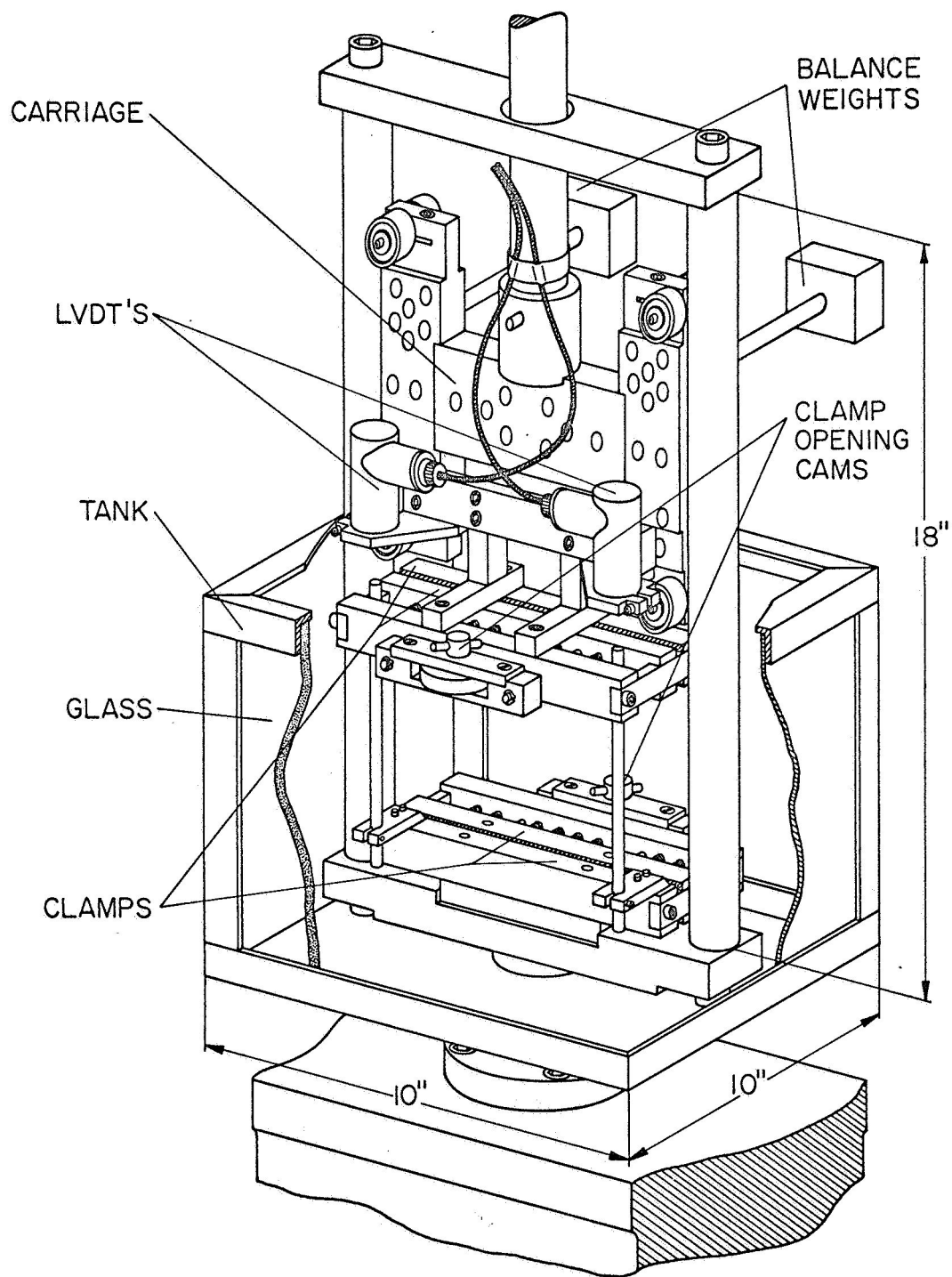


FIG. 3 DEVICE FOR THE
MECHANICAL CHARACTERIZATION
OF SWOLLEN SOLITHANE

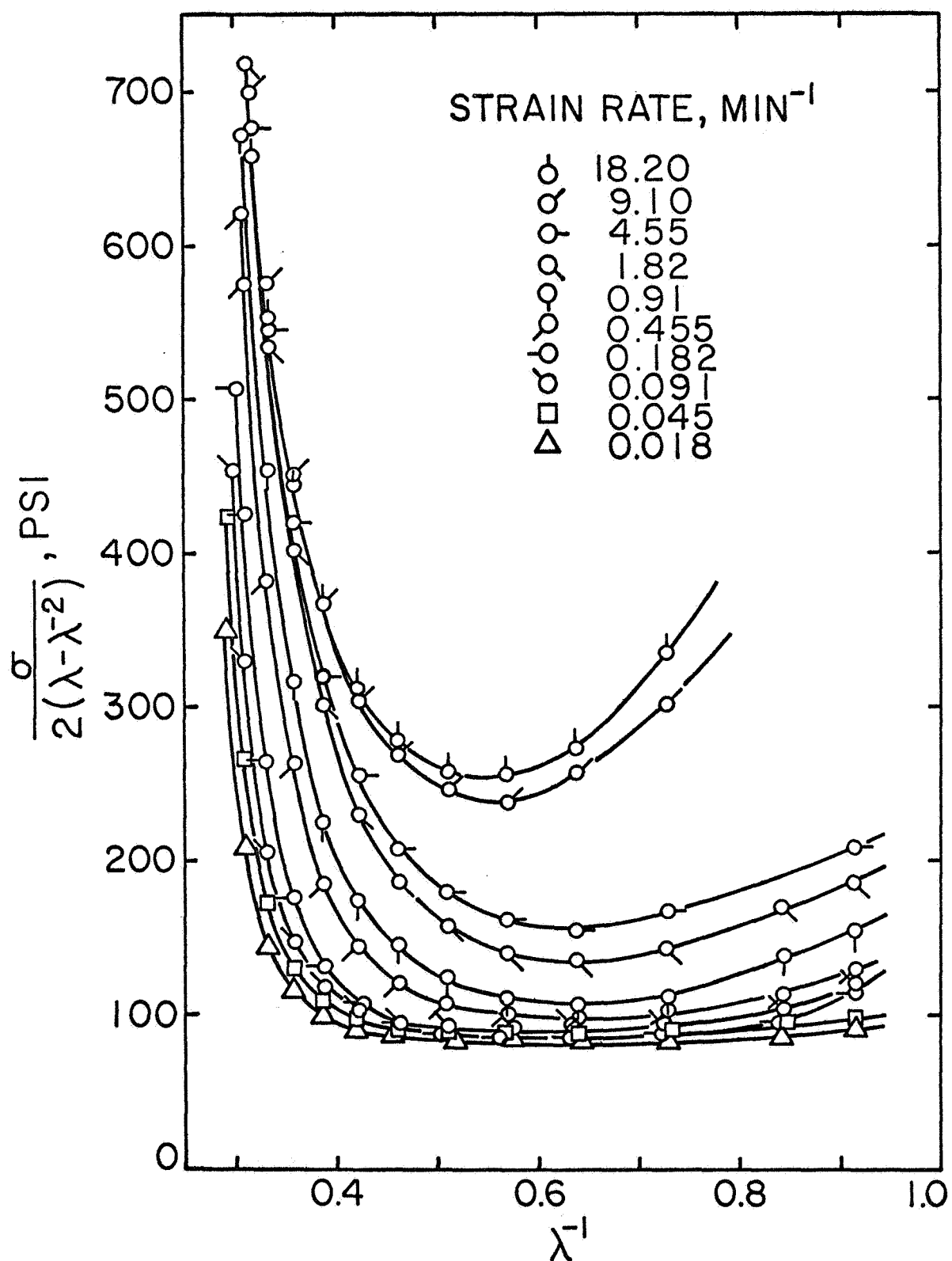


FIG. 4 MOONEY-RIVLIN PLOT FOR UNSWOLLEN SOLITHANE 50/50 AT -5 °C

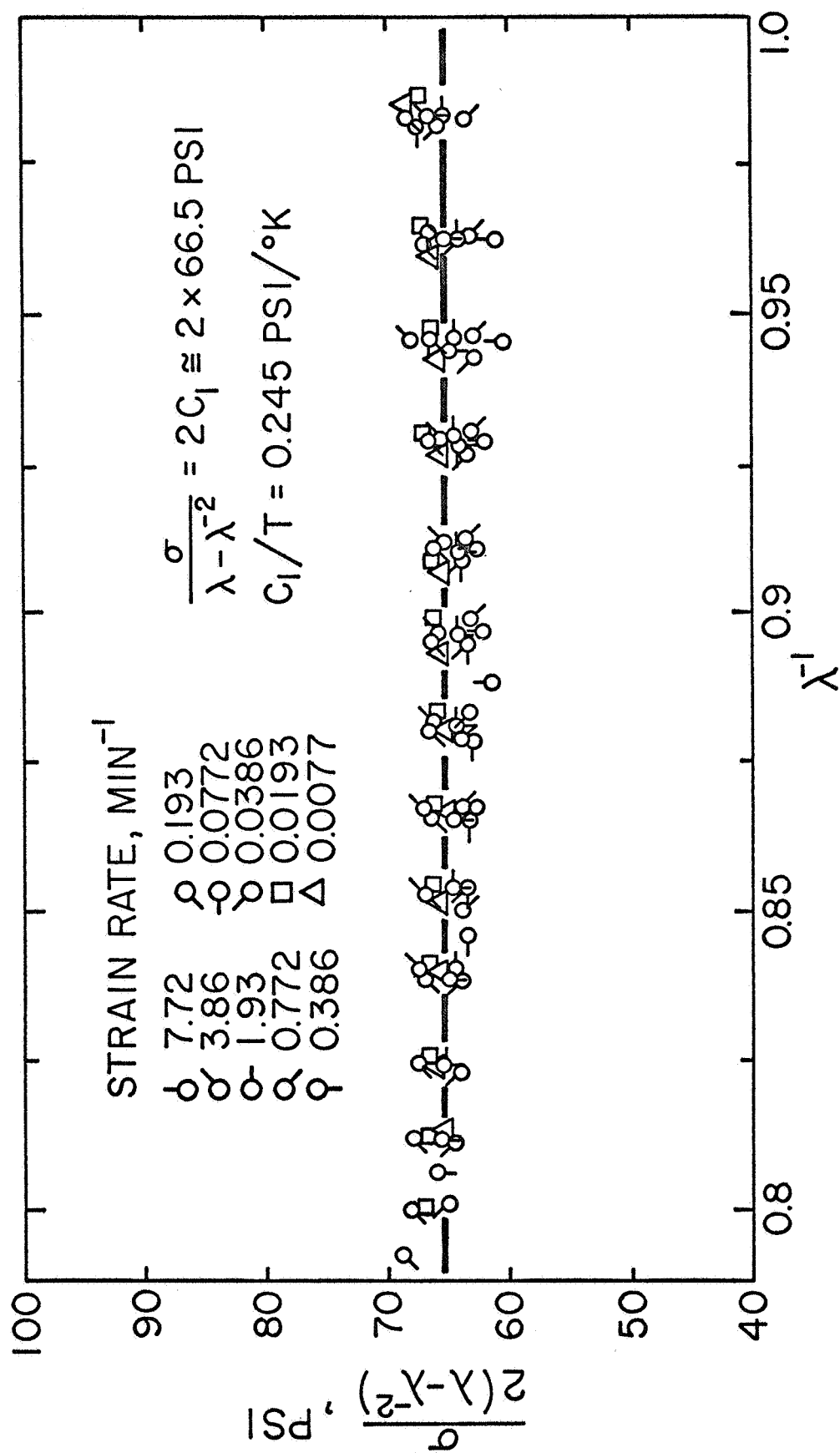


FIG. 5 MOONEY-RIVLIN PLOT FOR SOLITHANE 50/50 SWOLLEN IN TOLUENE AT -2°C

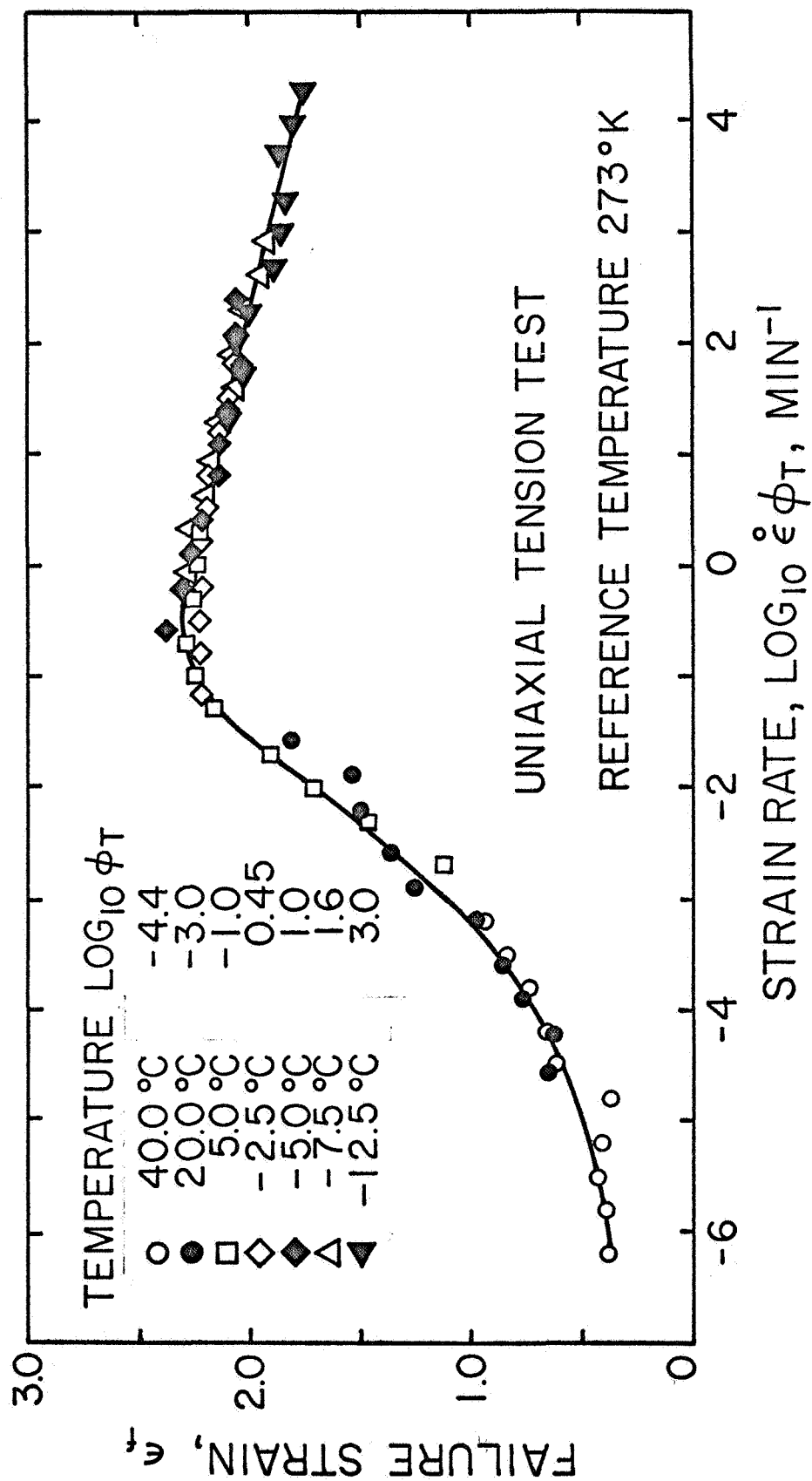


FIG.6 FAILURE STRAINS VS. REDUCED STRAIN RATE OF UNSWOLLEN SOLITHANE 50/50

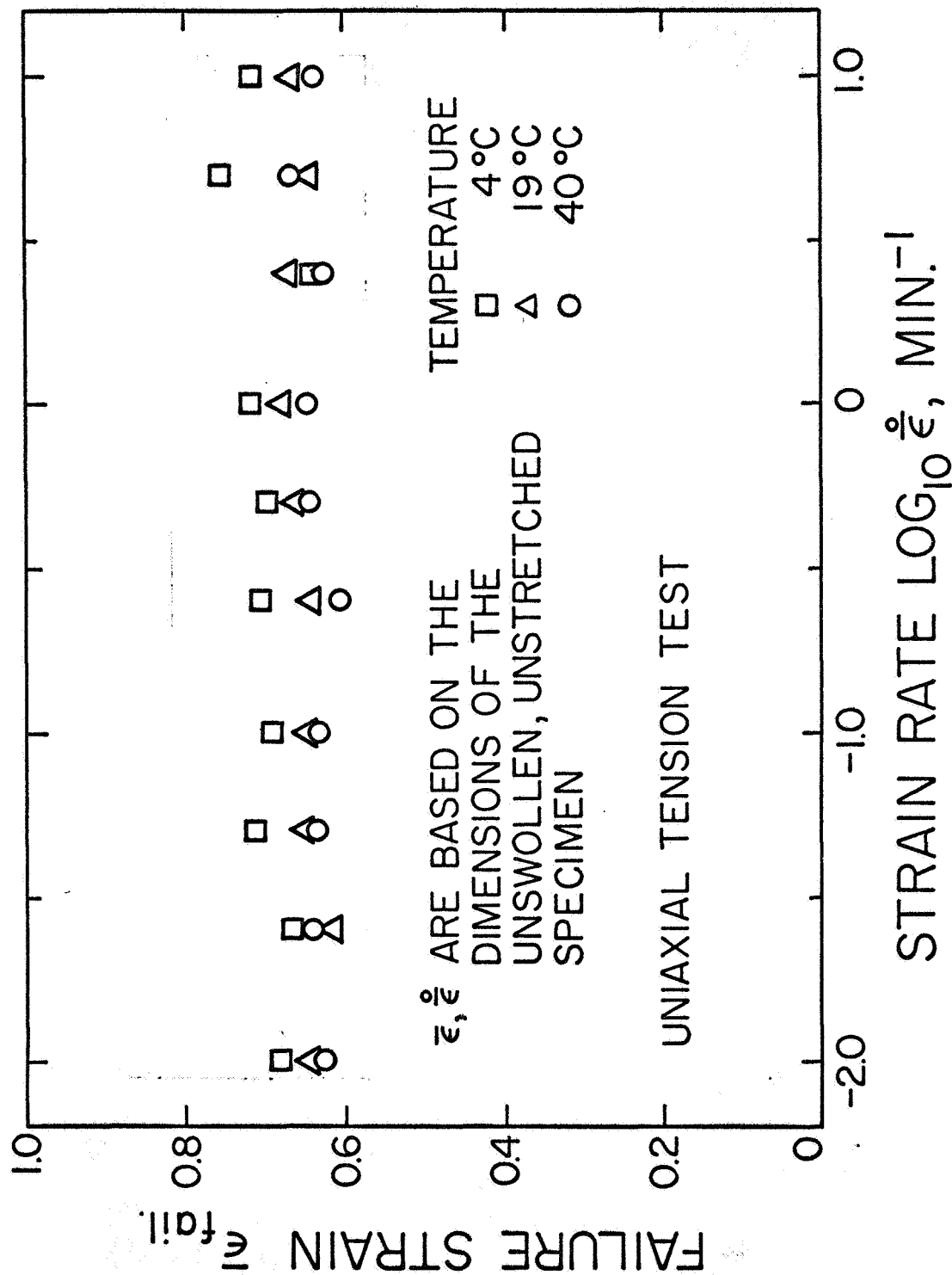


FIG. 7 FAILURE STRAIN VS. STRAIN RATE OF SOLITHANE 50/50 SWOLLEN IN TOLUENE

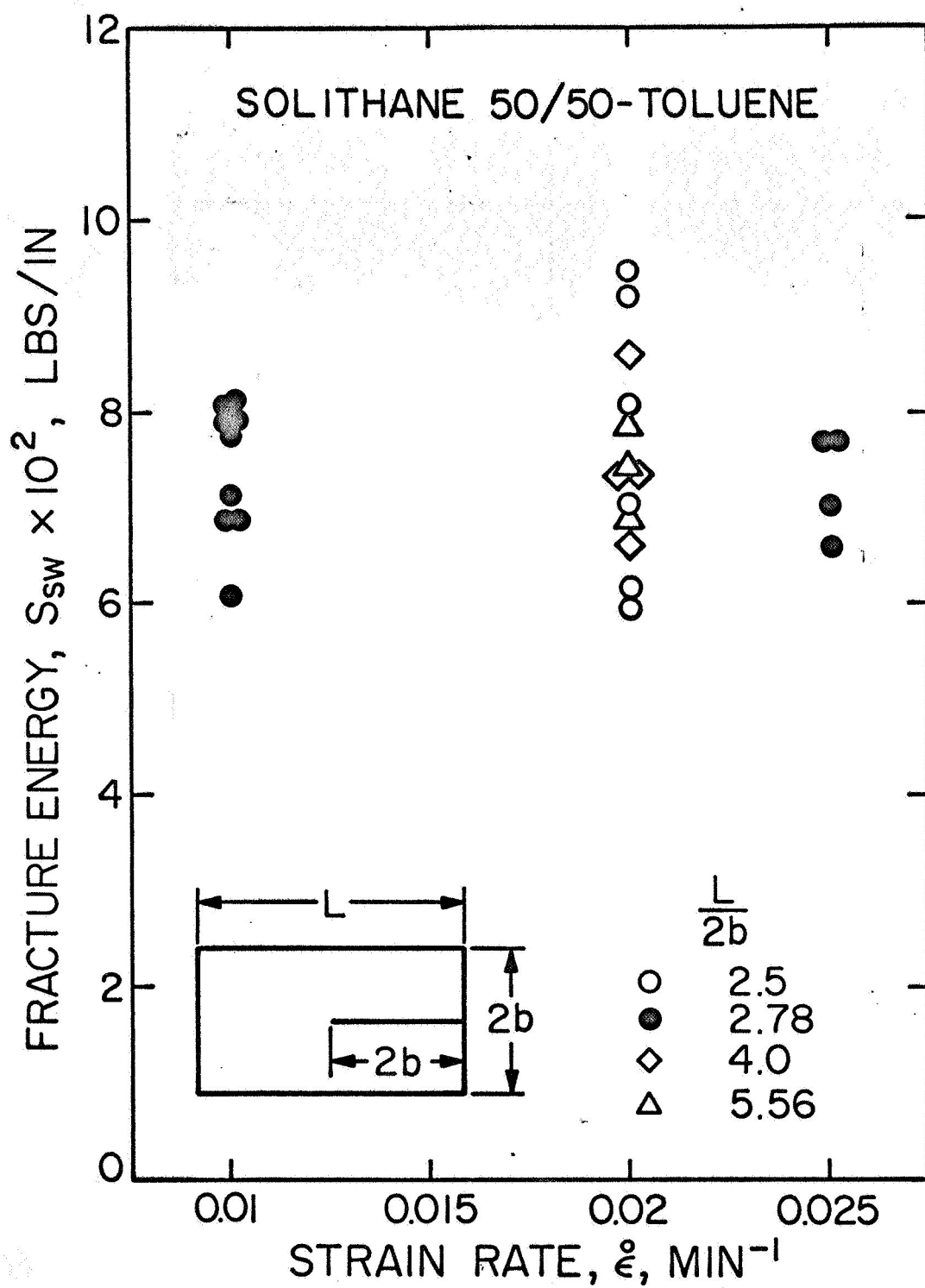


FIG. 8 FRACTURE ENERGY OF SWOLLEN SOLITHANE 50/50 AT 23 °C

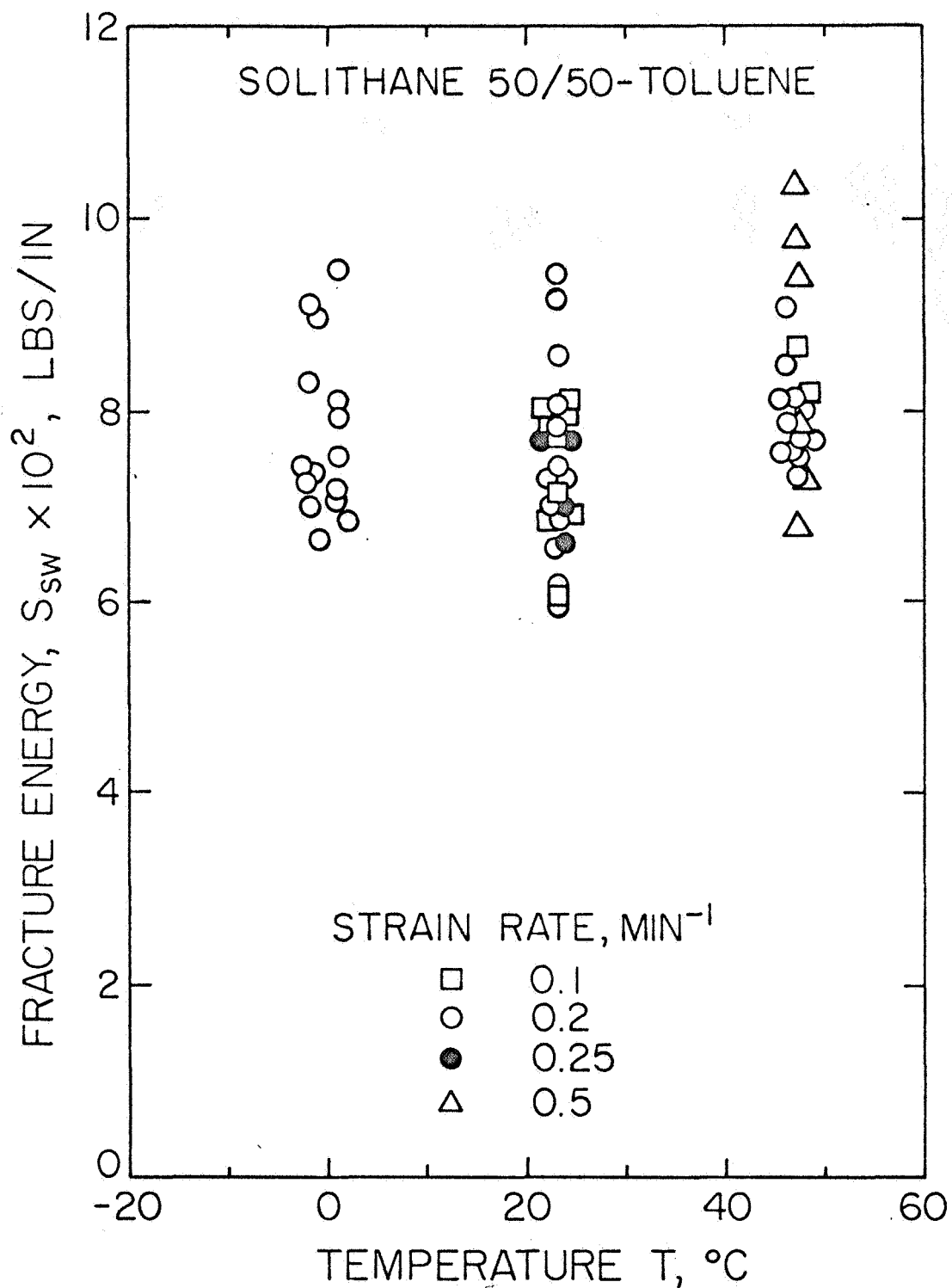


FIG.9 FRACTURE ENERGY OF SWOLLEN SOLITHANE 50/50 AS A FUNCTION OF TEMPERATURE

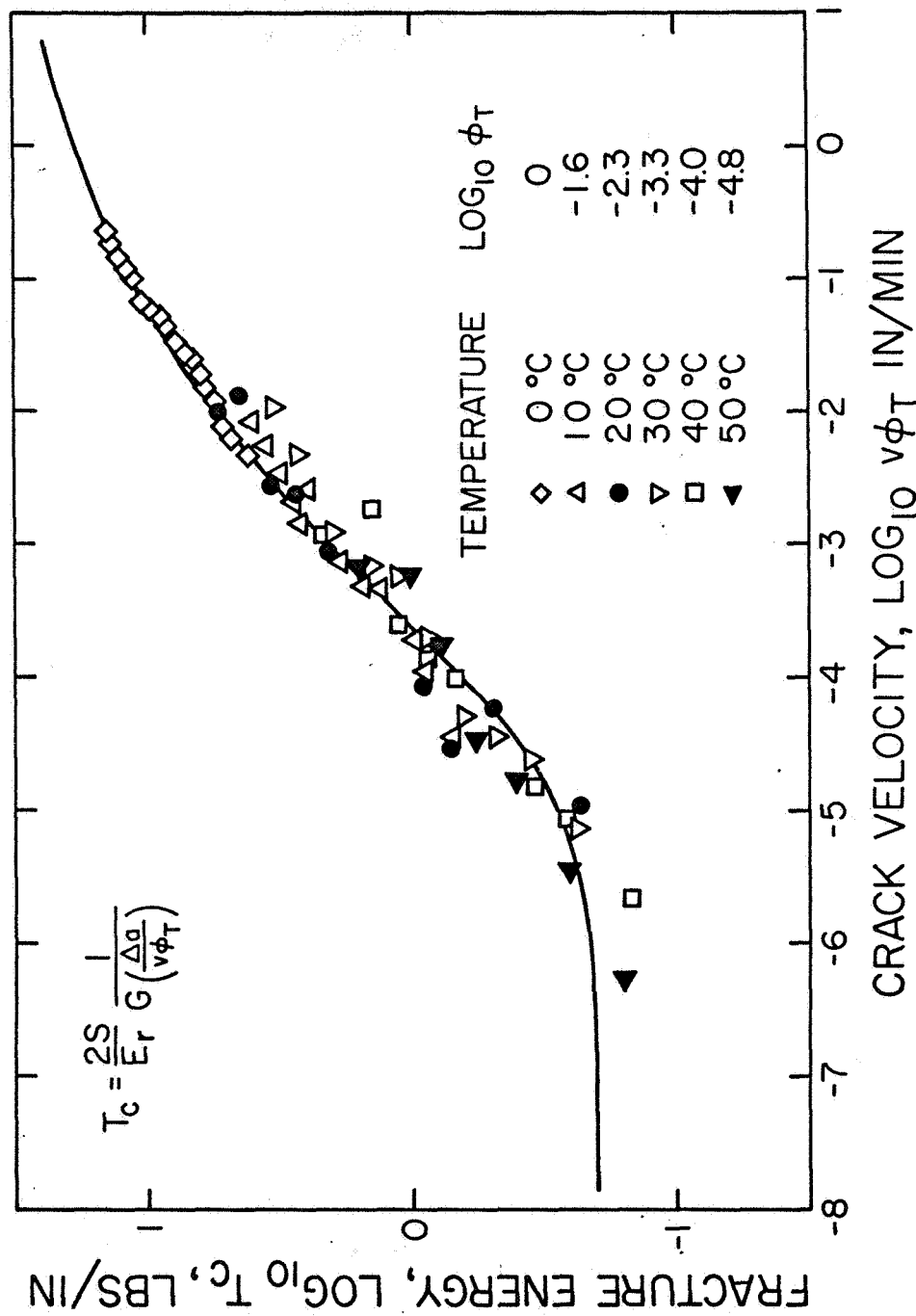


FIG. 10 FRACTURE ENERGY OF SOLITHANE 50/50 AS A FUNCTION OF CRACK VELOCITY AND TEMPERATURE

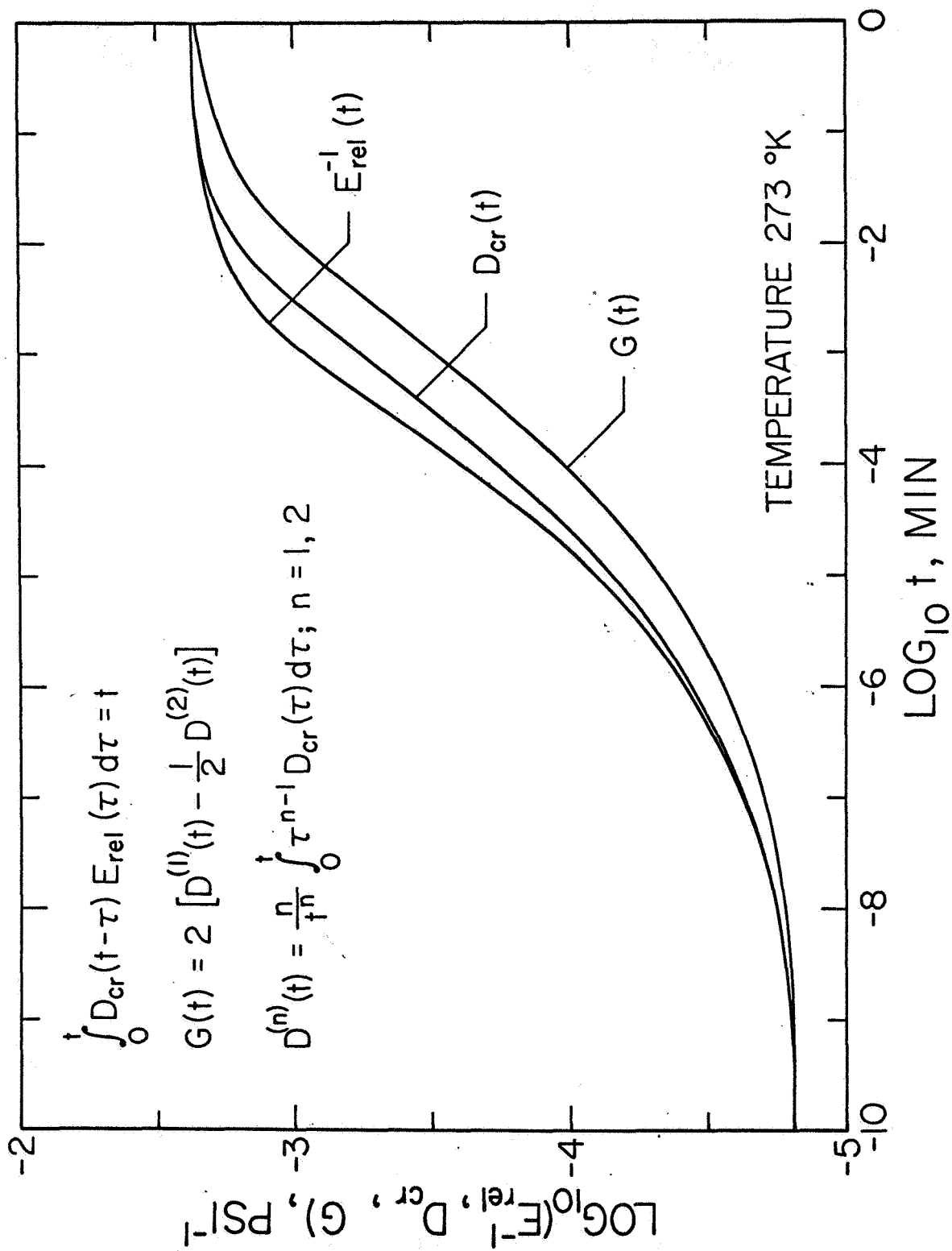


FIG. II THE RELAXATION FUNCTION, CREEP FUNCTION AND THE FUNCTION G FOR SOLITHANE 50/50